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U.S. DEPARTMENT OF ENERGY

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CHARACTERIZATION OF THE MICROSTRUCTURE OF Nb-1wt.%Zr-0.1wt.%C TUBES AS AFFECTED BY THERMOMECHANICAL PROCESSING

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Abstract

Microstructure of Nb-1Zr-0.1C tubes were characterized as affected by extrusion temperature of the tube shell and its thermomechanical processing to tubing. Two tube shells of about 40-mm outside diameter (OD) and 25-mm inside diameter (ID) were extruded 8:1 from a vacuum arc-melted ingot at 1900 and 1550 K. Two different OD tubes of ~0.36-mm wall thickness were fabricated from each tube shell by a series of 26 cold drawing operations with two in-process anneals. The microstructure of tube shells and the tubing before and after a 2-step heat treatment were characterized. Residue extracted chemically from each sample was also analyzed to identify the precipitates. The results concerning the effect of the initial extrusion temperature and subsequent processing on the microstructure of the tubes are presented together with a review of results from similar work on Nb-1Zr-0.1C sheet stock.

INTRODUCTION AND BACKGROUND

Nb-Zr alloys with and without carbon have been designated for advanced space power system applications based on their better resistance to liquid alkali metal corrosion and lower density as compared to other refractory metal alloys (Cooper 1984 and Kruger et al. 1989). A precipitate-hardened Nb-1Zr-0.1C (all compositions in wt.% unless otherwise indicated) alloy developed in '60s has been shown to be stronger than the solid solution-strengthened Nb-1Zr (Begley et al. 1963, DelGrosso et al. 1967 and Grigorovich and Sheftel 1982). A reactor test loop using both alloys have already been tested successfully for 3200 h (Bryhan and Chan 1993). Recent work (Titran 1990, Horak et al. 1991 and Uz and Titran 1993a) showed that (1) Nb-1Zr sheets with 0.06 and 0.1C have much better creep resistance than Nb-1Zr at 1350 K and (2) the creep resistance increased with increasing carbon content. The available data clearly indicate that a carbide-strengthened Nb-1Zr-C alloy must be used if a stress greater than 10 MPa is expected or a service life more than 2 years at 1350 K or above is required.

A thorough understanding of the effects of processing on the microstructure of Nb-Zr-C alloys is necessary because changes in the microstructure often cause changes in properties and performance. Studies on microstructural characterization, which date back to late '40s, (Norton 1949, Cuff 1962, Ostermann and Bollenrath 1966, Kissil et al. 1976, Grobstein and Titran 1986 and Uz and Titran 1991, 1993b) have established that the precipitates in Nb-Zr-C alloys are the orthorhombic Nb₂C and the cubic (Zr,Nb)C. Our recent work (Uz and Titran 1993c) on the long-term stability of the microstructure of Nb-1Zr-C sheet with and without applied stress reconfirmed these findings. Examination of sheet samples which were

creep tested for 10,000 - 34,500 h at 1350 K with a total creep strain of less than 0.3% showed that the microstructure of the Nb-1Zr-C alloys possessed excellent stability due to the finely distributed (Zr,Nb)C. These carbides, which are not present in the as-cast ingots, form by transformation of Nb₂C during processing and/or the subsequent high temperature exposure. The (Zr,Nb)C has a face-centered cubic (fcc) crystal structure with a lattice parameter (a_0) increasing from 0.450 to 0.468 nm with an accompanying increase in the Zr/Nb ratio. These a_0 values fall within the range of the a_0 for fcc-NbC (0.447 nm) and fcc-ZrC (0.470 nm) indicating that the cubic carbides detected in the Nb-1Zr-C alloys are solid solutions of NbC and ZrC. Once formed, the (Zr,Nb)C precipitates remain small ($\leq 1 \mu\text{m}$) and finely distributed throughout the matrix thus the Nb-1Zr-C alloys the excellent microstructural stability and creep resistance.

All the work cited on Nb-Zr-C alloys have been on cast and sheet products. However, because intended applications include tube forms, an investigation on the microstructure of Nb-Zr-C tubes is also important and necessary. This study characterizes the microstructure of thin-walled Nb-1Zr-0.1C tubes as affected by thermomechanical processing. In particular, the effects of the initial extrusion temperature (1900 and 1550 K) of the tube shell, subsequent drawing operations and a double-annealed heat treatment are examined. This study is part of an SP-100 project being carried out to optimize the process variables to obtain tubing with mechanical properties which exceed the design criteria of current advanced power systems.

EXPERIMENTAL

The condition of the materials examined in this study are given in Table 1. The Nb-1Zr-0.1C alloy was procured commercially in the form of a vacuum arc-melted (VAM) ingot with the specifications of 0.8-1.2 Zr, 0.075-0.1050 C, 0.015 O (max) and 0.01 N (max) with the balance being Nb. The fabrication of the tubes was performed at Oak Ridge National Laboratory (ORNL) under contract from National Aeronautics and Space Administration (NASA). Two tube shells, approximately 25 mm inside diameter (ID) by 37 mm outside diameter (OD), were extruded at an extrusion ratio of 8:1, one at 1900 K and the other at 1550 K (labeled 1- and 2-, respectively). Tubes with two different diameters (7.82 and 5.84 mm OD) were drawn from each tube shell. The drawing procedures were similar for both tube sizes with 26 cold draws and in-process anneals of 1 h at 1473 K following the 4th and 13th draws. These tubes (-26 and -26A in Table 1) differed in dimensions and slightly in percent cold work (%CW). Samples from all the tubes were double-annealed as the final heat treatment.

Samples from the ingot, tube shells and tubing were analyzed for chemical composition. Longitudinal cross-section (area parallel to the drawing axis) of the tube shells and tubing were examined in the as-polished condition by optical and scanning electron microscopy (SEM) to study the precipitate morphology and distribution. Samples from the double-annealed tubes were also examined in the as-polished and etched condition using optical microscopy to determine the grain size. The etchant used was a solution of lactic-nitric-hydrofluoric acids with a volume ratio of 6:3:1. A 900 ml methanol-100 ml bromine-10 g tartaric acid solution with Pt wire as catalyst was used to extract the precipitates

TABLE 1. Processing History of Nb-1Zr-0.1C Tube Shells and Tubes. All Tube Drawing Operations were Performed at Room Temperature.

MATERIAL	PROCESS/CONDITION	OD (mm)	ID (mm)	WALL (mm)
INGOT	As-cast vacuum arc-melted ingot			
1-AX ^(a)	Tube shell, as-extruded at 1900 K	36.83	25.40	5.72
1-26	As-drawn tube	7.82	7.11	0.36
1-26-DA ^(b)	1-26 after double-anneal	7.82	7.11	0.36
1-26A	As-drawn tube	5.84	5.08	0.38
1-26A-DA ^(b)	1-26A after double-anneal	5.84	5.08	0.38
2-AX ^(a)	Tube shell, as-extruded at 1550 K	36.58	26.16	5.21
2-26	As-drawn tube	7.82	7.11	0.36
2-26-DA ^(b)	2-26 after double-anneal	7.82	7.11	0.36
2-26A	As-drawn tube	5.84	5.08	0.38
2-26A-DA ^(b)	2-26A after double-anneal	5.84	5.08	0.38

^(a) The reduction ratio was 8:1 for all the extrusion operations.

^(b) Double Anneal, (2-step heat treatment of 1 h at 1755 K and 2 h at 1475 K).

from the samples as residue. The residues were analyzed by X-ray spectroscopy for the crystal structure and by an inductively-coupled plasma (ICP) technique for the Zr and Nb contents of the precipitates.

RESULTS AND DISCUSSION

Chemical Composition

The chemical analysis results in Table 2 show that the compositions of the initial ingot and the tubes were within the required specifications. The O and N impurity levels were low enough not to cause any concern, and all the tubes were very close to the nominal alloy composition of Nb-1Zr-0.1C.

Microstructure Characterization

Cast Ingot: The microstructure of cast ingots of Nb-1Zr-0.1C have been previously characterized (Uz and Titran 1993b,c) where they were found to consist of very coarse grains (up to 500 μm) with coarse ($>1 \mu\text{m}$) intra- and intergranular Nb₂C.

Tube Shells: The micrographs of tube shells extruded at 1900 K (1-AX) and 1550 K (2-AX) in Figure 1 shows that the effects of extrusion temperature on the precipitate morphology and distribution were quite distinct. The precipitates in 1-AX showed heavy alignment in the direction of extrusion, and varied in size from less than 1 μm to nearly 10 μm . The larger precipitates were mostly elongated and the smaller ones were more rounded-irregular.

TABLE 2. Chemical Compositions of Nb-1Zr-0.1C Ingot, Tube Shells and Tubes

SAMPLE	CHEMICAL COMPOSITION (wt.%)				GRAIN SIZE (μM)	ASPECT RATIO
	O ^(a)	N ^(a)	C ^(b)	Zr ^(c)		
INGOT	0.0050	0.0021	0.101	1.0		
1-AX	0.0045	0.0015	0.105	0.9		
1-26	0.0098	0.0024	0.099			
1-26A	0.0085	0.0019	0.100			
1-26-DA	0.0049	0.0026	0.101		23 \pm 2	1-6
1-26A-DA	0.0043	0.0029	0.101		28 \pm 2	1-6
2-AX	0.0058	0.0022	0.098			
2-26	0.0079	0.0022	0.098			
2-26A	0.0110	0.0010	0.100			
2-26-DA	0.0031	0.0041	0.097		31 \pm 2	1-6
2-26A-DA	0.0030	0.0074	0.103		32 \pm 6	1-6

^(a) Inert gas fusion, ^(b) Combustion extraction, ^(c) ICP method.

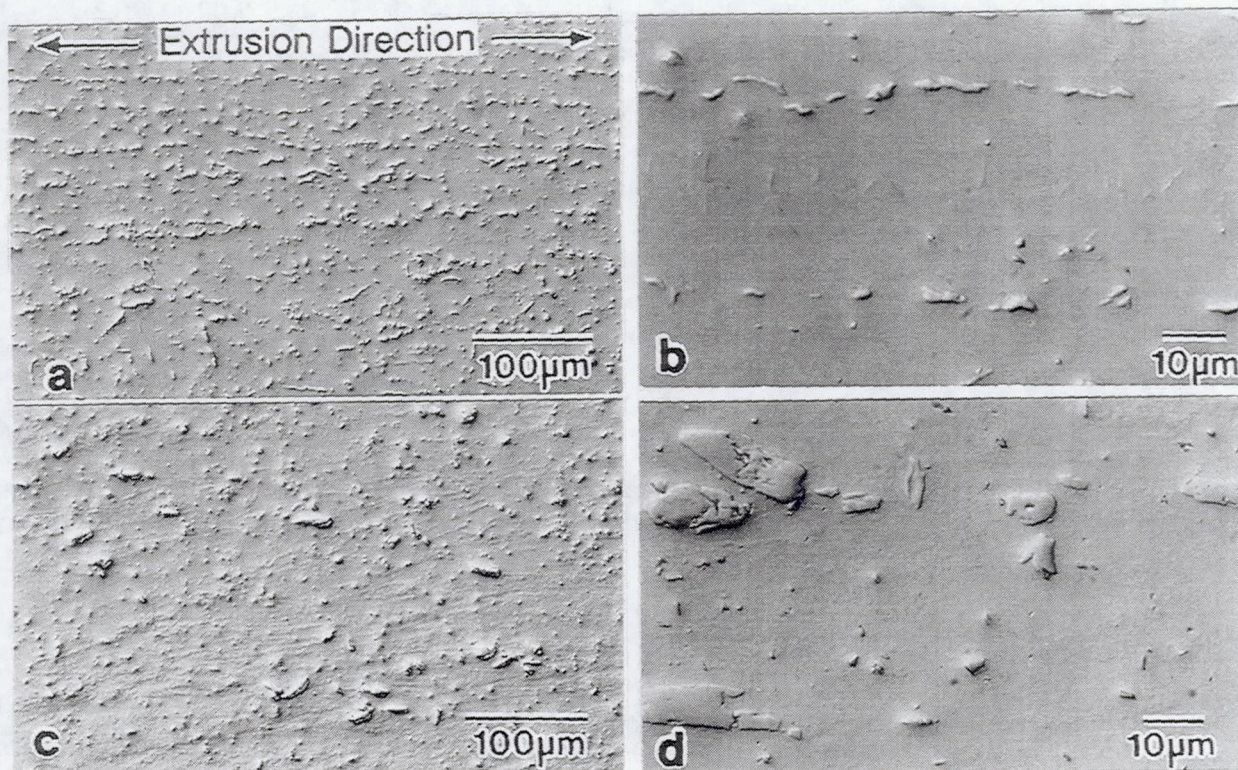


FIGURE 1. Optical Micrographs at Two Different Magnifications of the Tube Shells Extruded at 1900 K (a and b) and 1550 K (c and d). All As-Polished, Differential Interference Contrast (DIC).

Some needle-like precipitates were also present in the matrix. The precipitates in 2-AX were notably coarser than those in 1-AX. While submicron particles were present, some

precipitates were over 20 μm in diameter and fractured, possibly during extrusion. Alignment of precipitates along the extrusion direction was not readily evident; however, extensive deformation of the grains was visible. The finer precipitate size in 1-AX was expected because the solid solubility of C is higher at 1900 K than at 1550 K. This would lead to dissolution and subsequent re-precipitation of carbides to a larger extent during the higher temperature processing. Further evidence of the difference in microstructure was found by hardness testing where the Vickers hardness number (VHN_{100}) was about 131 ± 5 for 1-AX and 108 ± 5 for 2-AX. However, the effect of extrusion temperature was not as noticeable from the analysis of the residue from the tube shells (see Table 3). In both tube shells, the precipitates were predominantly Nb_2C , but some $(\text{Zr},\text{Nb})\text{C}$ was also detected. The Zr/Nb ratio, which was about 2/98 in the ingot, increased to about 10/90 in the tube shells indicating that processing at either temperature resulted in some transformation of Nb_2C to $(\text{Zr},\text{Nb})\text{C}$.

TABLE 3: Results of the Analysis of Phase-Extracted Residue of Nb-1%Zr-0.1%C Ingot, Tube Shells and Tubes.

SAMPLE	PHASE(S)	LATTICE PARAMETER ^(a) (nm)	CHEMICAL ANALYSIS ^(c) (Zr/Nb RATIO)
INGOT	Nb_2C ^(a)	-	2/98 (0.0204)
1-AX	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	- ^(b)	8/92 (0.0870)
1-26	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.451	10/90 (0.1000)
1-26A	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.459	35/65 (0.5385)
1-26-DA	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.456	30/70 (0.4286)
1-26A-DA	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.456	20/80 (0.2500)
2-AX	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.456	9/91 (0.0989)
2-26	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.459	40/60 (0.6667)
2-26A	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.460	30/70 (0.4286)
2-26-DA	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.459	30/70 (0.4286)
2-26A-DA	Nb_2C , $(\text{Zr},\text{Nb})\text{C}$	0.456	25/75 (0.3333)

^(a) All Nb_2C was orthorhombic with lattice parameters of $a_0 = 1.09$ nm, $b_0 = 0.3$ nm and $c_0 = 0.5$ nm.

^(b) Not enough peaks to determine lattice parameters.

^(c) The relative error in the ICP analysis of the residue was as high as 20% of each reported value because of the small amounts involved.

As-Drawn Tubes: There was no discernable microstructural difference between the as-drawn tubes as evidenced from Figure 2. The microstructure of each consisted of heavily deformed grains elongated in the drawing direction. The precipitates distributed throughout the matrix were around a micrometer in size with a few coarser particles. The similarity between the tubes -26 and -26A from each tube shell was expected, because the only difference between them was the size (Table 1). The similarity between the same-sized tubes from the two tube shells (1-26 and 2-26 or 1-26A and 2-26A in Table 1) indicates that the effect the extrusion temperature on the microstructure of the tube shell was nullified during the rather involved thermomechanical processing to tubing. The similarity in the microstructure of the tubes was reflected on their hardness values as well.

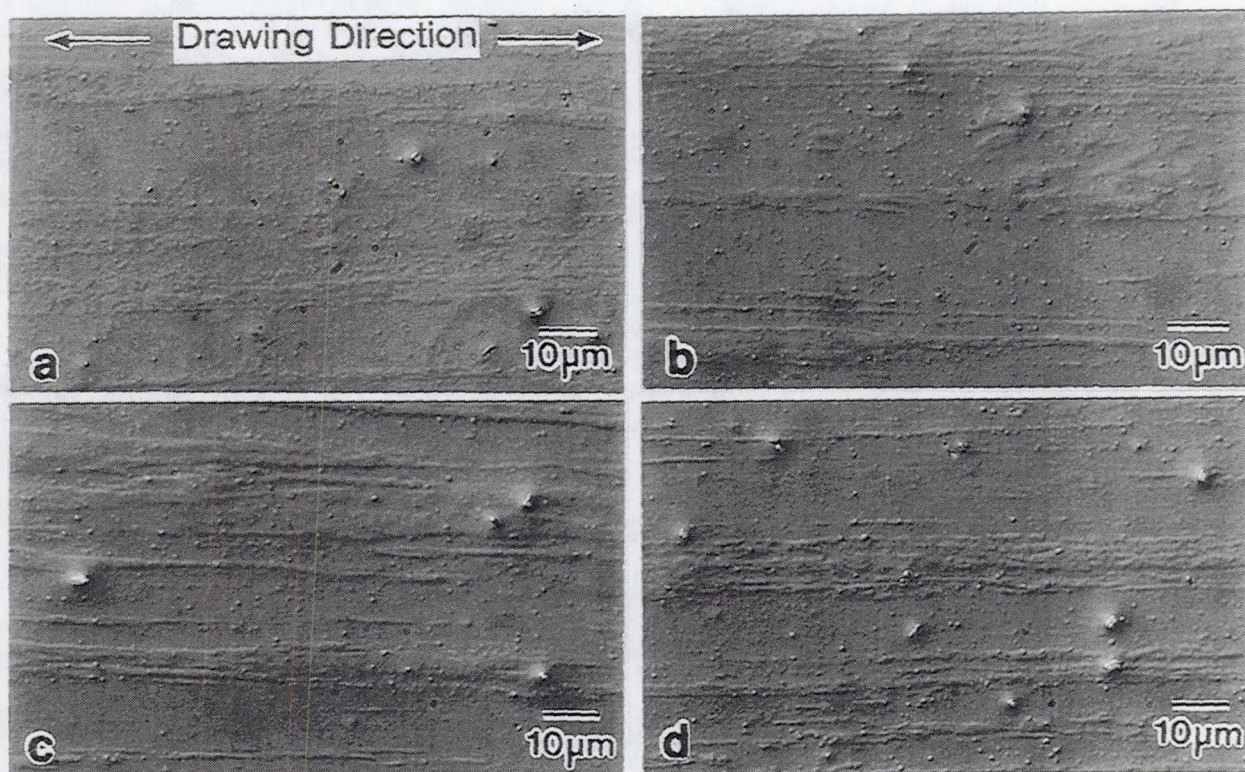


FIGURE 2. Optical Micrographs of 7.8-mm and 5.8-mm OD Tubes as Cold-Drawn from the Tube Shells Extruded at 1900 K (a and b) and 1550 K (c and d). All As-Polished, DIC.

The VHN_{100} was 147 ± 2 for the tubes from 1-AX and 146 ± 3 for those from 2-AX. As with the tube shells, the precipitates in the tubes were identified as Nb_2C and $(Zr,Nb)C$ (Table 3). However, the Zr/Nb ratio was higher for the tubes indicating further transformation of Nb_2C to $(Zr,Nb)C$ during the fabrication process.

Double-Annealed Tubes: Micrographs in Figure 3 show that the microstructures of the double-annealed tubes were also similar regardless of the extrusion temperature or tubing size. This was expected because of the similar microstructures prior to the heat treatment. The finely distributed precipitates were mostly $1 \mu m$ in size with a few about $5 \mu m$. Again, the precipitates were identified as Nb_2C and $(Zr,Nb)C$ in all the tubes with no significant change in the Zr/Nb ratio upon double-annealing. (Table 3). The grain size was measured employing a lineal analysis method (ASTM Standard E112 1984) on polished and etched sections (Figure 4). The average grain size was similar in each case and the average grain diameter varied from ~ 25 to $30 \mu m$ with an aspect ratio of less than 6 (Table 2). While the equiaxed grains were as small as $10 \mu m$ in diameter in all, the long axis of the largest grains measured $\sim 150 \mu m$ in 1-26-DA and 1-26A-DA, and nearly $300 \mu m$ in 2-26-DA and 2-26A-DA. The microhardness, VHN_{100} , of the tubes from 1-AX was 96 ± 5 , and it was 90 ± 5 for the tubes from 2-AX, which are quite lower than the values for the as-drawn tubes. These, once more, fall within about one standard deviation of one another, and reflect the similarity in microstructure. The protrusions in the DIC micrographs (Figs. 1-4) were identified as the carbides based on the analysis of phase-extracted residue samples.

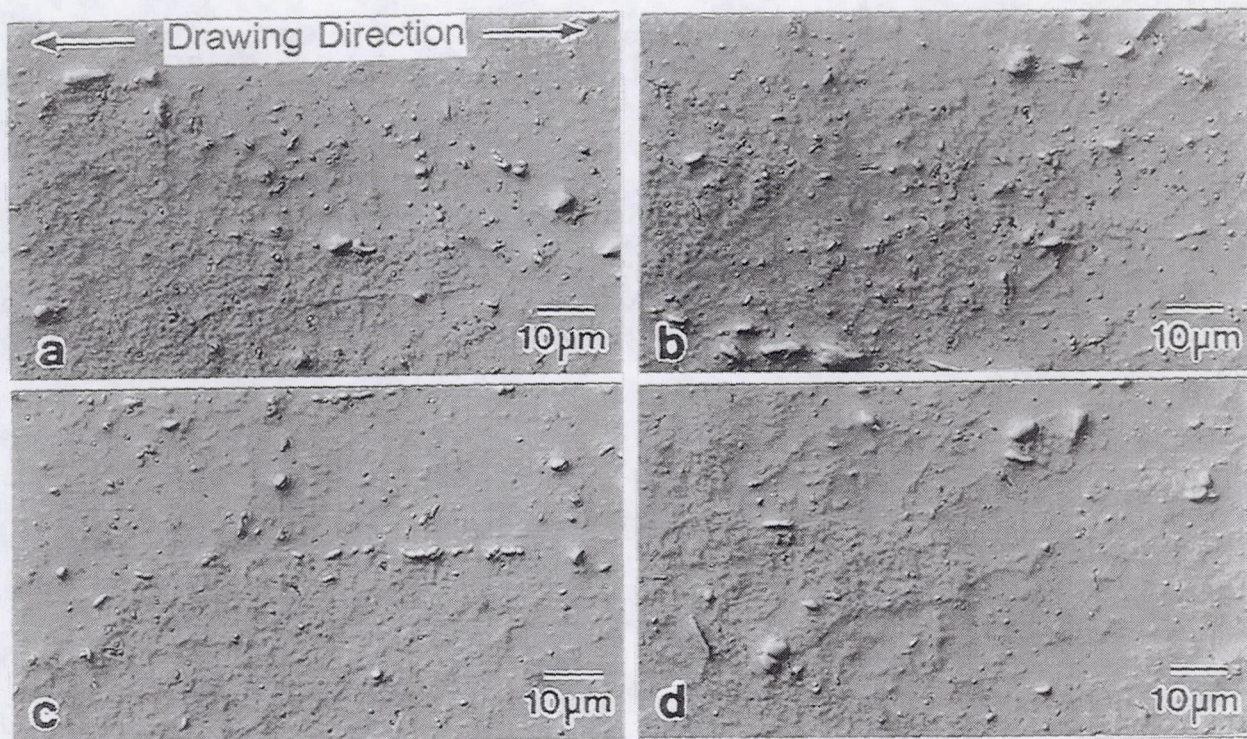


FIGURE 3. Optical Micrographs of Double-Annealed 7.8-mm and 5.8-mm OD Tubes from the Tube Shells Extruded at 1900 K (a and b) and 1550 K (c and d). All As-Polished, DIC.

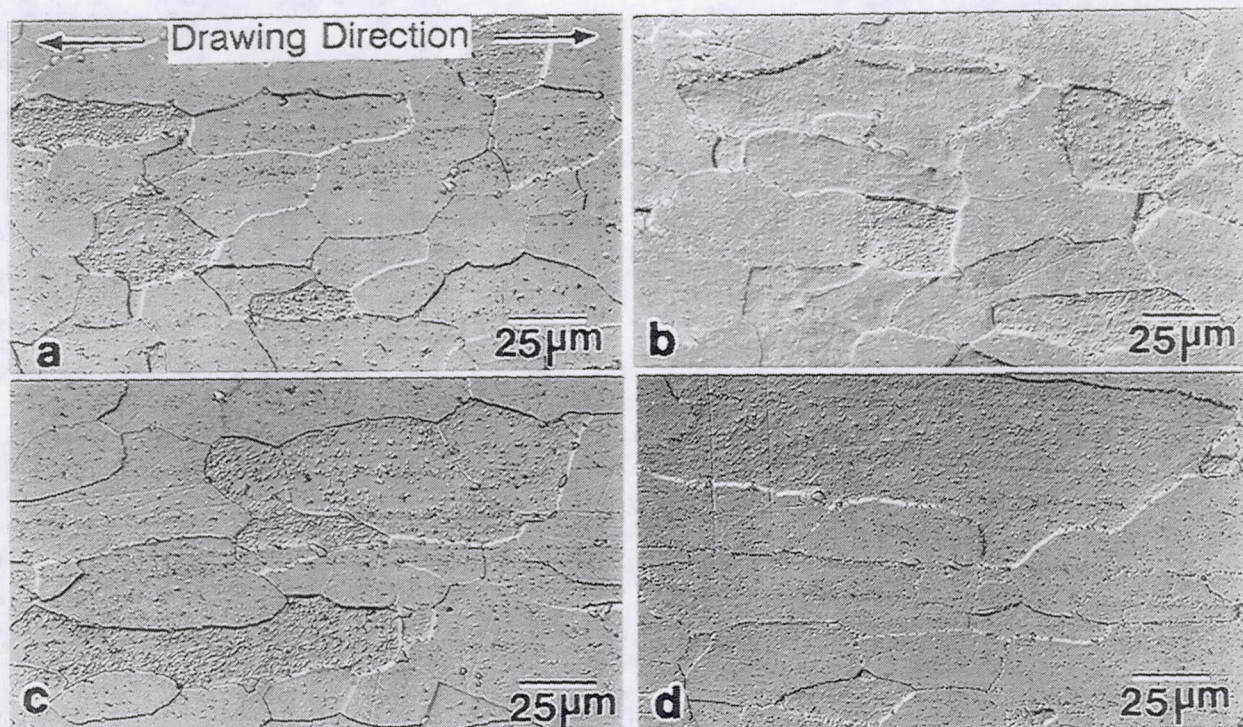


FIGURE 4. Optical Micrographs of Double-Annealed 7.8-mm and 5.8-mm OD Tubes from the Tube Shells Extruded at 1900 K (a and b) and 1550 K (c and d). All As-Polished and Chemically Etched, DIC.

To further verify that the protrusions were not optical artifacts, we examined the samples using SEM in both the secondary and back-scattered electron modes (SE and BSE, respectively). Typical SEM micrographs are shown in Figure 5: (a) and (b) shows, respectively, SE and BSE images of the same area from 1-26A-DA, while (c) and (d) are BSE images of similar regions from 1-26-DA and 2-26-DA, respectively. These SEM photomicrographs also show the similar morphology and distribution of the precipitates in all the tubes. In the BSE images, the large gray particles are Nb_2C and the small gray ones are the cubic carbides with the Nb-Zr matrix appearing lighter than the carbides because of its larger average atomic number. The black regions are pores or voids from carbide pull-out.

The results from the as-drawn and double-annealed tubes in this study are similar to those we obtained on sheet in an earlier study (Uz and Titran 1993a). They most resemble the results on a Nb-1Zr-0.1C sheet fabricated by cold rolling a sheet bar following triple 4:1 extrusions at 1900 K. This sheet had a VHN_{100} of about 158 ± 7 in the as-rolled condition with about 60% cold work, and 104 ± 5 after double annealing heat treatment. In the double-annealed condition, its grains had an average diameter of $32 \pm 3 \mu\text{m}$ and 1-10 aspect ratio. This sheet had excellent microstructural stability and creep resistance (less than 0.3% total creep strain in about 10,000 h at 1350 K and 34.5 MPa). Based on its steady state creep rate, this sheet was estimated to reach 2% total creep strain in over 12 years. In an ongoing creep test at NASA Lewis Research Center, a sample from the same sheet has undergone

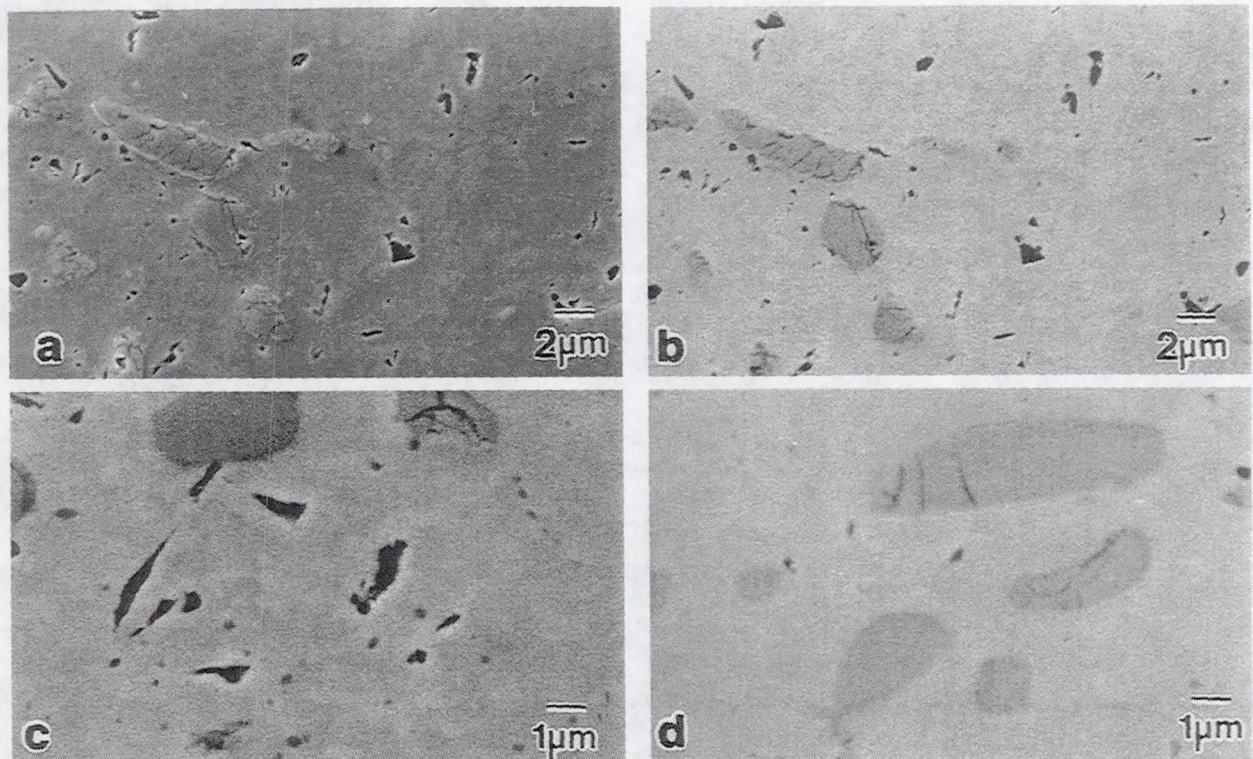


FIGURE 5. SEM Micrographs of 5.8-mm Tubes from the Tube Shell Extruded at 1900 K (a - c) and 7.8-mm Tube from the Tube Shell Extruded at 1550 K (c). All in Double-Annealed Condition and As-Polished. (a) Secondary Electron Image, (b-d) Back-Scattered Electron Images.

less than 0.3% strain after over 13,000 h at 1450 K and 24 MPa. Currently, there are no other test data available to us for these tubes except for the microstructural studies reported here. However, we would expect the tubes to have as good a creep resistance as the sheet based on their similarities in microstructure.

SUMMARY OF RESULTS AND CONCLUSIONS

We studied the effects of initial extrusion temperature and the subsequent thermomechanical processing on the microstructure of Nb-1Zr-0.1C tubes. An 8:1 extrusion at 1900 K yielded a tube shell with a finer microstructure than the same at 1550 K. Both extrusions resulted in break-down of the as-cast grain structure and some transformation of Nb₂C to the stable cubic carbide (Zr,Nb)C. The as-drawn and double-annealed tubes from either extrusion were similar in both microstructure and microhardness indicating that the effect of the extrusion temperature on the tube shells was not transmitted to the final tubing from them.

The results of this study on tubes is consistent with those we obtained on sheets in that thermomechanical processing resulted in refinement of the microstructure and transformation of Nb₂C to extremely stable (Zr,Nb)C. The microstructure of all the tubing resembles that of a Nb-1Zr-0.1C sheet (with a much different processing history than the tubes) which we showed to have excellent high temperature stability and creep resistance. This would indicate that materials with similar microstructure and properties can be obtained by different processing schedules.

Based on our experience with sheets and the results of this work, we conclude that the subsequent processing rather than the initial extrusion conditions dictated the properties of the final Nb-1Zr-0.1C sheet and tubing. However, more mechanical property testing of the tubes is needed to confirm the premise that the tubes have similar properties to the alloy sheet.

Acknowledgements

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13. ABSTRACT (Maximum 200 words) Microstructure of Nb-1Zr-0.1C tubes were characterized as affected by extrusion temperature of the tube shell and its thermomechanical processing to tubing. Two tube shells of about 40-mm outside diameter (OD) and 25-mm inside diameter (ID) were extruded 8:1 from a vacuum arc-melted ingot at 1900 and 1550 K. Two different OD tubes of ~0.36-mm wall thickness were fabricated from each tube shell by a series of 26 cold drawing operations with two in-process anneals. The microstructure of tube shells and the tubing before and after a 2-step heat treatment were characterized. Residue extracted chemically from each sample was also analyzed to identify the precipitates. The results concerning the effect of the initial extrusion temperature and subsequent processing on the microstructure of the tubes are presented together with a review of results from similar work on Nb-1Zr-0.1C sheet stock.				
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